INCH-POUND

MIL-PRF-25604E
01 October 97
Superseding
MIL-P-25604D
23 May 1969

PERFORMANCE SPECIFICATION
PROPELLANT, uns-DIMETHYLHYDRAZINE

This specification is approved for use by all
Departments and Agencies of the Department of Defense

1. SCOPE

1.1 Scope. This specification covers the requirements for unsymmetrical dimethylhydrazine $(CH_3)_2NNH_2$ propellant (UDMH).

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements documents cited in sections 3 and 4 of this specification, whether or not they are listed.

2.2 Government documents.

2.2.1 Specifications, standards, and handbooks. The following specifications form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those listed in the issue of the Department of Defense Index of Specifications and Standards (DoDISS) and supplement thereto, cited in the solicitation (see 6.2).

SPECIFICATIONS

FEDERAL

BB-H-886 - Hydrogen
BB-A-1034 - Air, Compound, for Breathing Purposes

DEPARTMENT OF DEFENSE

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Code (68) SA-ALC/SFSP, 1014 Billy Mitchell Blvd/STE 1, Kelly AFB TX 78241-5603, by using the standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A FSC 9135

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.

MIL-PRF-27401 - Propellant Pressurizing Agent, Nitrogen

MIL-PRF-27407 - Propellant Pressurizing Agent, Helium

(Unless otherwise indicated, copies of the above specifications, and standards are available from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia PA 19111-5094).

2.3 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DoDISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DoDISS are the issues of the documents cited in the solicitation (see 6.2).

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D 1193 - Specification for Reagent Water (DoD adopted)

ASTM D 1298 - Standard Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method (DoD adopted)

ASTM D 2276 - Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling (DoD adopted)

ASTM E 29 - Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications (DoD adopted)

(Application for copies should be addressed to the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken PA 19428-2959.

2.4 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Chemical and physical properties. The chemical and physical properties of the propellant shall conform to those listed in Table I when tested in accordance with the applicable test methods.

3.2 Limiting values. The following applies to all specified limits in this specification: For purposes of determining conformance with these requirements, an observed value or a calculated value shall be rounded off “to the nearest unit” in the last right-hand digit used in expressing the specification limit according to the rounding-off method of ASTM Practice E 29 for using Significant Digits in Test Data to Determine Conformance with Specifications.

3.3 Filter. A filter with a 10-micrometer nominal and 40-micrometer absolute rating shall be installed between the manufacturer's plant system and the container to be filled for delivery.

3.4 Qualitative. The propellant shall be colorless, homogeneous liquid when examined visually by transmitted light.

TABLE I. Chemical and physical properties.

PROPERTIES	LIMITS	TEST PARAGRAPHS
uns-Dimethylhydrazine (Percent by weight)	98.0 min	4.3.2
Water (Percent by weight)	0.3 max	4.3.2
Amines (Percent by weight)	1.5 max	4.3.2
N-Nitrosodimethylamine (Percent by weight)	0.01 max	4.3.6
Chloride (Percent by weight)	0.003 max	4.3.4
Density (g/mL) @ 60°F	0.795 to 0.797	4.3.5
Particulate (mg/L)	10 max	4.3.3

4. VERIFICATION

4.1 Classification of inspections. The inspections shall be classified as quality conformance inspections.

4.2 Quality conformance inspection. Unless otherwise specified (6.2), each filled shipping container shall be considered a lot and shall be sampled. Each sample shall be subjected to the visual examination described in 4.3.1 for conformance to 3.4 and to the tests described in Table I for conformance to the requirements specified in Table I.

4.2.1 Sample. A sample consists of not less than 600 mL of propellant. Unless otherwise specified, quality conformance tests shall be made on the sample of propellant taken directly from the shipping container. When required, the sample shall be forwarded to a laboratory designated by the procuring activity for subjection to the quality conformance tests specified herein. The bottle intended for sampling shall be specially cleaned and handled according to the procedure described in 4.3.3

4.2.2 Rejection. When any sample of the propellant tested in accordance with 4.3 fails to conform to the requirements specified herein, the entire lot represented by the sample shall be rejected.

4.3 Test methods.

4.3.1 Examination of product. The propellant shall be visually examined while performing test specified in 4.3.3 to determine compliance with the requirement as specified herein. Examination to ensure that the material conforms to 3.4 shall be conducted after the sample has been transferred to the 500 mL calibrated cylinder.

4.3.2 Uns-Dimethylhydrazine assay, water and amines. The propellant, water, and amine content of the sample shall be determined by the following method.

4.3.2.1 Gas chromatographic method.

4.3.2.1.1 Assay column preparation. Weigh 5 grams of polyethylene glycol 400 and 45 grams of 60/80 mesh Fluoropak 80 into separate beakers. Dissolve the polyethylene glycol 400 in a volume of reagent grade dichloromethane which is approximately one-half the volume of the Fluoropak 80. Pour the Fluoropak 80 into the polyethylene glycol 400 solution with gentle stirring. Spread the resulting damp powder in a tray and dry the mixture in a vacuum oven at 100°C and less than 50 mm of Hg for at least one hour.

Cap one end of a 1/8 inch OD by 6-foot stainless steel tube and fill the tube with the prepared column packing by pouring through a small funnel attached to the other end. Tap or mechanically vibrate the tube to ensure uniform packing. When the tube is filled, plug both ends with a small wad of glass wool, bend the column to the configuration required by the column oven, and connect the column to the inlet fitting in the oven. Condition the column with carrier gas flowing and the oven set at ~120°C for one hour. After conditioning the column, connect the other end to the detector and set the carrier gas flow to approximately 25 mL/min, and the column oven to 100°C.

The inlet and detector temperatures, if separately heated, shall be set to 100°C and 150°C, respectively. The detector current should be set to a nominal sensitivity value recommended for helium by the instrument manufacturer.

4.3.2.1.2 Analysis. Equilibrate the column with propellant by injection of two or more 5 microliter (µL) samples into the inlet. If more than 30 minutes elapse between analyses, a single 5 µL injection of propellant should re-equilibrate the column. Inject 1 - 2 µL of propellant for analysis and record the areas of all peaks in the chromatogram. Each analysis should require less than 15 minutes for elution of all components. The elution order of possible sample components is as follows: Air, ammonia, methylamine, dimethylamine, formaldehyde dimethylhydrazone, UDMH, water, methylhydrazine, and hydrazine.

4.3.2.1.3 Calculations. The following formula shall be used to calculate the percent by weight of each component appearing in the chromatogram.

$$\%C = \frac{A_c}{\sum A_i} \times 100$$

where

A_c = The measured area of a peak multiplied by its signal attenuation factor.

ΣA_i = The sum of all the measured areas multiplied by their respective signal attenuation factors.

%C = The weight percent of the component corresponding to A_c.

Assumption: The thermal conductivities of all components in the sample are equal.

4.3.2.1.4 Equipment and reagents. The following equipment and reagents shall apply as test conditions of 4.3.2.

a. Equipment:

(1) Gas chromatograph - incorporating a thermal conductivity detector.

(2) Recorder - potentiometric strip chart, 0 - 1 millivolt, 1 second full scale response, with integrator.

(3) Tubing - stainless steel, 1/8 inch O.D. x 6 feet

(4) Hypodermic syringe - 10 microliter, fixed needle

(5) Regulator - helium, to fit the cylinder

(6) Standard screens - 60 mesh and 80 mesh

(7) Vacuum oven, capable of 100°C

b. Reagents:

(1) Fluoropak 80, Analabs, Inc., 80 Republic Dr., North Haven CT 06473 or equivalent.

(2) Polyethylene glycol 400, or equivalent

(3) Dichloromethane - ACS reagent grade

(4) Helium gas - conforming to MIL-P-27407

4.3.3 Particulate. The propellant sample shall be tested for contamination in accordance with ASTM D-2276, Method A, with the following exceptions:

4.3.3.1 Mix the sample thoroughly without exposure to air. Immediately pour 500 mL of the sample into a clean 500-ml graduated cylinder. Use this 500 mL of propellant for the particulate analysis.

4.3.3.2 Use a solvent resistant filter disc made from such materials as Millipore LSWP-04700 (Mitex-Teflon), Millipore URWP 04700, (Solvinert) or Gelman VF-6, (Fluoride-Metricel), plain, white, 10±3 microns, 47 mm diameter, or equivalent.

4.3.3.3 The drying oven temperature shall be 158°F (70°C) instead of the 194°F (90°C) specified in ASTM D-2276.

4.3.3.4 Filtered isopropyl alcohol shall be used for rinsing the sample bottle and filter holder instead of petroleum ether specified in ASTM D-2276.

4.3.4 Chloride.

4.3.4.1 Procedure. Evaporate 2.0 mL of propellant and 0.2 mL of 1N sodium hydroxide contained in a 25 mL volumetric flask using a steambath or hot plate located in a fume hood. Purge the flask with nitrogen gas to facilitate evaporation. Dissolve the residue with 2.0 mL of ferric ammonium sulfate reagent and 2 mL of chloride-free distilled water. Add 1.0 mL of saturated mercuric thiocyanate reagent, mix by swirling, and dilute to the 25 mL mark with chloride-free distilled water. Mix again by inverting the flask several times and allow to stand in darkness for 15 to 30 minutes. Measure the absorbance of the reagent blank and sample solutions at 460 nm in a 5.0-cm cell, after setting distilled water to "0" absorbance. Subtract the absorbance of the reagent blank from the sample absorbance. The chloride content is determined from the calibration curve constructed according to 4.3.4.3.

4.3.4.2 Calculation. Calculate the percent chloride in the sample using the following formula:

$$Cl^{-}, weight\% = \frac{(mg\ Cl^{-}\ from\ calibration\ curve)}{1.57} \times 10^{-1}$$

4.3.4.3 Calibration Curve. A calibration curve shall be prepared as follows: Syringe 0 mL, 1 mL, 2 mL, 4 mL, and 8 mL of the dilute chloride standard solution (0.010 mg/mL) into separate 25 mL volumetric flasks. Add 0.20 mL of 1N sodium hydroxide to each flask. Add 2.0 mL ferric ammonium sulfate and 5 mL chloride-free water and swirl. Add 1.0 mL saturated mercuric thiocyanate and mix. Add chloride-free distilled water to the 25 mL mark and mix by inverting several times. Place standards and blank in darkness for 15 to 30 minutes. Measure the absorbance of the blank and each of the four standards in a 5.00 cm cell at 460 nm after setting distilled water to "0" absorbance. Subtract the absorbance of the blank from that of each standard. Plot this difference against the corresponding mg of Cl .

4.3.4.4 Reagents and equipment.

4.3.4.4.1 Reagents.

a. Sodium hydroxide solution, 1N: Dissolve 40 grams of ACS reagent grade sodium hydroxide pellets in 300 mL of chloride-free distilled water contained in a 1000 mL volumetric flask. Cool the solution to room temperature, dilute to the mark with the chloride-free distilled water, and mix thoroughly.

Transfer the solution to a plastic bottle and protect from prolonged exposure to the atmosphere.

b. Ferric ammonium sulfate: Mix 240 mL concentrated nitric acid and 160 mL of chloride-free water. Dissolve 48.2 grams of ferric ammonium sulfate, ACS grade, in the nitric acid solution and allow the precipitate to settle out. Decant the liquid for use in the analysis.

c. Saturated mercuric thiocyanate: Saturate ACS reagent grade ethyl alcohol (95%) with ACS reagent grade mercuric thiocyanate. Allow the excess to settle out and decant the supernatant liquid for use in the analysis.

d. Chloride stock solution: Dissolve exactly 165 mg (0.165g) of dried, primary standard grade, sodium chloride in chloride-free distilled water contained in a 100 mL volumetric flask and dilute to the mark (1.0 mg of Cl^-/mL).

e. Chloride standard solution: Syringe exactly 10.0 mL of the Chloride stock solution into a 1000 mL volumetric flask, and dilute to the mark with chloride-free water (0.010 mg Cl^-/mL).

f. Chloride-free water: This shall be double-distilled, or distilled deionized water, which tests negative (no cloudiness) with silver nitrate.

4.3.4.4.2 Equipment.

a. Syringes: 1, 2, and 10 mL capacity

b. Volumetric flasks: 25-, 100-, and 1000-mL capacity

c. Reagent bottles as necessary.

d. Steam bath, or hot plate.

e. Analytical balance.

f. Spectrophotometer or filter photometer: Single or double beam capable of accommodating 5.0 cm cells.

4.3.5 Density. The density of the propellant shall be determined in accordance with ASTM D 1298, using the hydrometer calibrated at 25°C.

4.3.6 N-Nitrosodimethylamine. The N-nitrosodimethylamine (NDMA) content of the propellant shall be determined as follows:

4.3.6.1 Column Preparation. Weigh 10 grams of Alltech AT-220 (formerly Amine 220) and 20 grams of support material into separate beakers. Dissolve the AT-220 in a volume of reagent grade dichloromethane which is approximately equal to the volume of the support material. Pour the support material into the AT-220 solution with gentle stirring, spread the resulting damp powder in a tray, and dry the mixture in a vacuum oven at 100°C and less than 50 mm of Hg for at least one hour.

Cap one end of a 1/8 inch OD by 6-foot stainless steel tube and fill the tube with the prepared column packing by pouring through a small funnel

attached to the other end. Tap or mechanically vibrate the tube to ensure uniform packing. When the tube is filled, plug both ends with a small wad of glass wool, bend the column to the configuration required by the column oven, and connect the column to the inlet fitting in the oven. Condition the column with carrier gas flowing and the oven set at ~150°C for one hour. After conditioning the column, connect the other end to the detector and set the carrier gas flow to approximately 30 mL/min, and the column oven to 115°C. The inlet and detector temperatures, if separately heated, shall be set to 120°C and 150°C, respectively. The hydrogen and air flowrates should be set to those recommended by the instrument manufacturer.

4.3.6.2 Analysis. Inject 1 - 3 microliters of the calibration standard into the chromatograph and record the area of the NDMA peak (retention time = approximately 8 min). Inject the same volume of propellant and record the area of the NDMA peak.

4.3.6.3 Calibration Standard. Place 0.10 mL of NDMA (0.10 grams) into a 1000 mL volumetric flask containing ~500 mL of distilled water, mix, and dilute to the mark. The calibration standard contains 0.1 micrograms of NDMA per microliter of solution.

4.3.6.4 Calculation. Calculate the results as follows:

$$\%NDMA = \frac{A \times S}{0.785 \times V} \times 10^{-1}$$

where: A = area of NDMA peak in sample chromatogram multiplied by the signal attenuation.

$$S = \frac{0.1 \times volume\ of\ standard\ injected\ (microliters)}{Area\ of\ standard\ NDMA\ peak \times signal\ attenuation}$$

V = volume of sample injected (microliters)

4.3.6.5 Reagents and Equipment. The following shall apply as test conditions of 4.3.6:

a. Reagents

(1) Support Material. White diatomaceous earth. Acid and Base washed, silanized, 70/80 mesh.

(2) Alltech AT-220, Alltech Associates,Inc, 2051 Waukegan Road, Deerfield IL 60015-1899, or equivalent.

(3) Dichloromethane, ACS Reagent Grade.

(4) Helium Gas, conforming to MIL-P-27407.

(5) Hydrogen Gas, conforming to BB-H-886.

(6) Breathing Air, conforming to BB-A-1034.

b. Equipment

(1) Gas chromatograph, equipped with a flame ionization detector.

(2) Recorder, potentiometric, strip chart, 0 - 1 millivolt span, 1 second full scale response with integrator (mechanical or electronic).

(3) Tubing, 1/8 inch OD x 6 feet, stainless steel.

(4) Hypodermic syringe, 10 microliter, fixed needle.

(5) Regulators, to fit cylinders of helium, hydrogen, and air.

(6) Vacuum oven, capable of 100°C

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of materiel is to be performed by DoD personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Department or Defense Agency, or within the Military Department's System Command. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful but is not mandatory.)

6.1 Intended use. The propellant described by this specification is intended for use as an fuel in rocket engines.

6.2 Acquisition requirements. Acquisition documents must specify the following:

a. Title, number, and date of the specification.

b. Issue of DoDISS to be cited in the solicitation, and, if required, the specific issue of individual documents referenced (see 2.2.1 and 2.3).

c. Method of shipment, type and capacity of containers.

d. Quantity by weight.

e. When a different sampling plan is required (4.2).

f. Packaging requirements (see 5.1).

6.3 Part or identifying number (PIN). The PIN to be used for this propellant is M25604.

6.4 Subject term (key word listing).

Fuel
uns-Dimethylhydrazine
N-Nitrosodimethylamine
Propellant
Rocket engine

6.5 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

Custodians
Navy - AS
Air Force - 68

Preparing Activity
Air Force - 68

Review Activities
Air Force - 19

Civil Agency Interest
NASA

Project No. 9135-0142

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.
2. The submitter of this form must complete blocks 4, 5, 6, and 7.
3. The preparing activity must provide a reply within 30 days from receipt of the form.

NOTE: This form may not be used to request copies of documents, nor to request waivers, or clarification of requirements on current contracts. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements.

I RECOMMEND A CHANGE:	1. DOCUMENT NUMBER	2. DOCUMENT DATE (YYMMDD)
	MIL-PRF-25604E	971001

3. DOCUMENT TITLE

Propellant, uns-DIMETHYLHYDRAZINE

4. NATURE OF CHANGE *(Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.)*

5. REASON FOR RECOMMENDATION

6. SUBMITTER

a. NAME (Last, First, Middle Initial)	b. ORGANIZATION	
c. ADDRESS (Include Zip Code)	d. TELEPHONE (Include Area Code) (1) Commercial (2) AUTOVON (If applicable)	7. DATE SUBMITTED (YYMMDD)

8. PREPARING ACTIVITY

a. NAME	b. TELEPHONE (Include Area Code)	
SA-ALC/SFSP	(1) Commercial (210) 925-7847	(2) AUTOVON (210) 925-7847
c. ADDRESS (Include Zip Code) 1014 Billy Mitchell Blvd, STE 1 Kelly AFB, TX 78241-5603	IF YOU DO NOT RECEIVE A REPLY WITHIN 45 DAYS, CONTACT: Defense Quality and Standardization Office 5203 Leesburg Pike, Suite 1403, Falls Church, VA 22041-3466 Telephone (703) 756-2340 AUTOVON 289-2340	